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Cesium Gettering by Graphite

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19. ABSTRACT *(Continue on reverse if necessary and identify by block number)*
In cesium (Cs) beam tube (CBT) clocks, only a small fraction of the Cs atoms effusing from the oven travel through the microwave cavity. The remaining fraction is gettered by pieces of graphite and graphite-coated surfaces found at various locations inside the clock. Should the graphite getters cease absorbing Cs, the increasing Cs background would rapidly obscure the true signal, resulting in rapid reduction in the signal-to-noise ratio. Getter failure is then one of the primary life-limiting processes in the Cs beam tubes. We are investigating the gettering properties of graphite in order to be able to characterize the type and the amount of graphite needed to efficiently getter Cs. From a microscopic angle we study the sticking coefficient of Cs on graphite as a function of time, under conditions similar to that encountered in a beam tube. A significant experimental observation is the rapid decrease in the sticking coefficient from an initial value of 1.0 to 0.25-0.30 after several days of exposure of Cs. We are also investigating the bulk gettering properties using various analytical techniques. Of particular interest is the maximum amount of Cs a given graphite can absorb. Preliminary measurements show that graphite can absorb approximately 20% by weight.

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I. INTRODUCTION/BACKGROUND

In the cesium (Cs) beam tube clocks (CBT) only a small fraction of the Cs atoms effusing from the oven travel through the microwave cavity where they undergo the 9192.6 MHz microwave transition. The remaining fraction of the effusing Cs atoms are collimated and gettered by slits and baffles made of graphite placed at various locations inside the clock. This is schematically shown in Fig. 1. The inner surfaces of the beam tube are also coated with graphite (Aquadag) to improve the gettering. Should the graphite getters cease absorbing Cs, the increasing background of Cs atoms would rapidly obscure the true signal needed to operate the standard reliably. The clocks will also be rendered unusable if the insulators in the electric feedthroughs get shorted by the conductive coatings of the background Cs atoms. Getter failure is then a life-limiting process in the Cs clock. For the clocks to be reliable, the gettering lifetime should be at least as long as the guaranteed lifetimes of the tubes. Standard failure is of particular concern in satellite applications, such as GPS, where reliable operation over the specified tube lifetime, 7.5 years, is required.

The practice of putting graphite blocks as getters inside the beam tubes dates back to the early days of the development of Cs beam frequency standards. Although the usefulness of graphite as a Cs getter has all along been qualitatively recognized, no systematic study of the Cs gettering properties of graphite has so far been reported. We have been experimentally investigating the gettering properties of graphite in order to be able to reliably characterize the type and amount of graphite to be used in space-borne clocks.

Many materials are effective surface and bulk getters (Ref. 1). In surface getters the gas molecules collide and react with the surface. There is little diffusion into the material. In bulk getters the efficiency is determined by the diffusion of the impinging molecules into the bulk. In both cases, the characteristics of the surface and the interaction between the bombarding atoms and the surface should be such that the incident atoms stick

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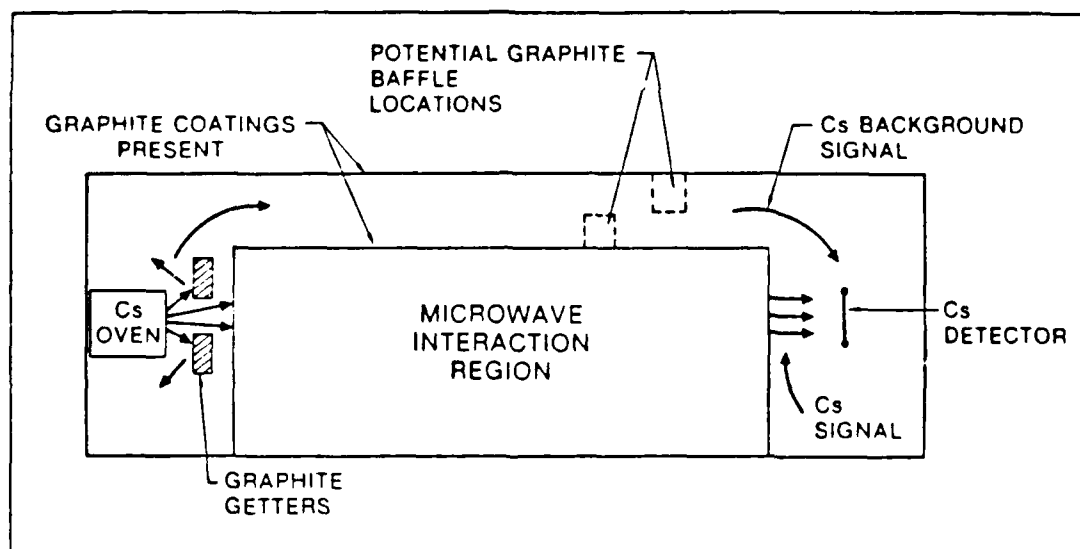**Cs GETTERING IN THE BEAM TUBE CLOCK**

Fig. 1. Schematic Representation of a Cs Beam Tube, Showing the Locations of Graphite Getters. The exposed metal surfaces inside the tube are also coated with Aquadag to further reduce the migration of Cs atoms to the detector region.

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well to the surface. Stated quantitatively, the prerequisite for efficient gettering is that the sticking coefficient γ be close to 1. Therefore, study of the sticking coefficient enables one to address the problems of gettering from a microscopic view. Another approach is to study the bulk gettering properties of graphite, e.g., how much Cs a particular weight of graphite may absorb. In our laboratory we are investigating from both the microscopic (sticking coefficient experiments) and the macroscopic (bulk gettering) approaches.

In the second section we present the details of our experimental apparatus for measuring the sticking coefficient of Cs on graphite. The dependence of the sticking coefficient on the sample preparation procedures is also discussed. In the third section the experimental results of our bulk gettering studies are presented. The implications of our experimental findings is discussed in the final section.

II. EXPERIMENTAL DETAILS AND DATA

A. MEASUREMENT OF STICKING COEFFICIENT (γ)

To measure the sticking coefficient γ of Cs on graphite we use an atomic beam apparatus. The experimental technique is illustrated in Fig. 2. A well-collimated beam of Cs atoms effusing from an oven is directed towards a graphite target. The reflected beam at a particular angle θ is measured using a surface ionization detector. We measured the sticking coefficient of Cs on graphite relative to a paraffin-coated surface, which is known to be perfectly nonsticking. The sticking coefficient γ is defined as

$$\gamma(\theta) = 1 - \frac{\text{reflected signal from graphite at } \theta^{\circ}}{\text{reflected signal from paraffin at } \theta^{\circ}}$$

We are studying the evolution of γ as a function of time for various incident Cs atomic beam intensities. Typical background pressure in the atomic beam apparatus is $\sim 1 \times 10^{-8}$ torr.

Graphite samples used in the present experiments are first baked externally in a quartz tube furnace at $1000^{\circ}C$ under liquid-nitrogen-trapped rough vacuum for a minimum of two hours, by which time the residual gas pressure is found to drop to a few microns. Glass seals are used everywhere in order to prevent the contamination of graphite samples. The inner walls of the quartz oven are thoroughly cleaned before the start of baking. The graphite sample is then loaded into a copper holder inside the atomic beam apparatus and pumped down to $\sim 1 \times 10^{-7}$ torr. The sample is baked to $350^{\circ}C$ in high vacuum for several hours until the background pressure is below 1×10^{-7} torr. The vacuum chamber containing the graphite target is kept isolated from the source chamber containing the Cs oven, except while measuring the reflected beam signal. This is accomplished by placing a gate valve between the two chambers. Physical isolation is necessary because even at room temperature we detected a weak Cs beam effusing from the oven. Inadequate and improper baking of the graphite samples gets reflected in the rapid decrease
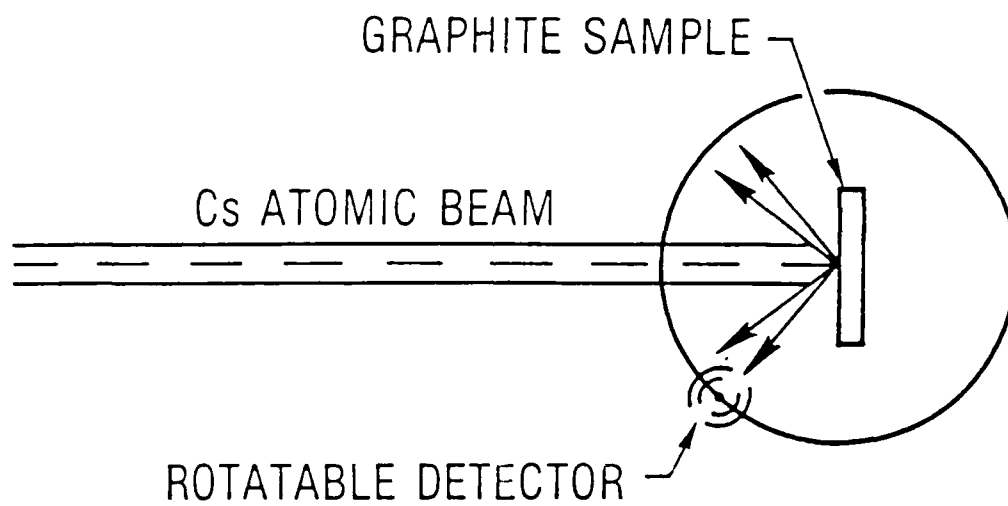
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Fig. 2. Experimental Arrangement for Measuring the Sticking
Coefficient γ of Cs On Graphite.

of the Cs sticking coefficient. When properly degassed, graphite can be made more gas-free than most common metals used in high vacuum systems. It is generally believed that graphite degassed at 1000 - 1100°C is "well outgassed." (Ref. 2)

The residual gas pressure in our apparatus is usually less than 1×10^{-8} torr. This is accomplished primarily by baking the vacuum chambers at about 100°C for about 24 hours. This improvement in the high vacuum resulted in a marked decrease in the background signal recorded by the surface ionization detector. Under best operating conditions, the typical background signal is $< 10^{-15}$ A whereas the reflected Cs signal at 70° is $\sim 10^{-11}$-10^{-12}A. The collision rate of the background gas at the residual gas pressure of 1×10^{-8} torr is ~ 27 monolayers hour, which is comparable to the collision rate of Cs on the graphite target. It is conceivable that what we are observing is actually a gas-phase reaction catalyzed by the surface--in other words, reaction of the impinging Cs atoms with the residual gas atoms on the surface. The evidence counter to this is the fact that our experimental data essentially remains unchanged when the Cs beam flux is increased by a factor of 2 and residual gas pressure is reduced by about a factor of 2.

1. Short-Cs Exposure Times

Our experimental results are shown in Fig. 3. Cs beam intensities range from 1×10^{12} to 4×10^{12} atoms/cm^2 sec. These intensities are low compared to what would be seen by a getter placed directly in front of the oven in a CBT, but comparable to intensities observed by graphite pieces placed at other locations in the beam tube. We have studied different types of graphite--POCO CZR-2, TRA-2, and Ultra Carbon UT6ST; all are known to have high porosity which should lead to superior gettering properties. Our experimental data showed excellent reproducibility. For the short duration of six hours, no significant difference in behavior was observed for the different types of graphite studied. It is clear from our data that the rate of decrease of the sticking coefficient with time is nearly the same for the three different intensities of the incident Cs beam. However, care must be exercised in extrapolating our data to long exposure times.
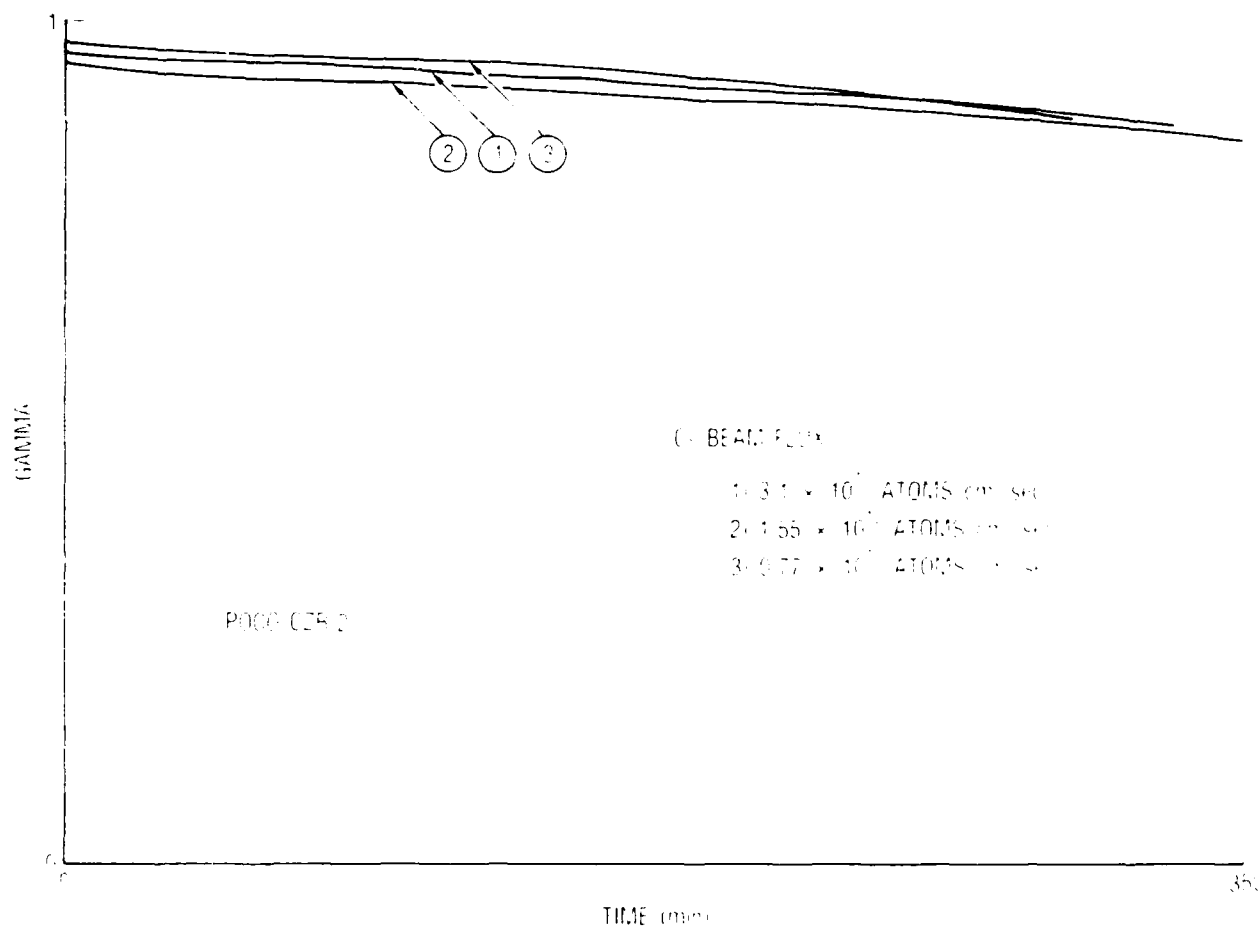
Fig. 3. Experimental Data for γ vs t Obtained After Improvements and
Modifications Made in the Apparatus. γ is essentially the same for
three different Cs beam intensities.

One of the factors that plays a crucial role in ensuring a high gettering efficiency is proper baking of the graphite samples. We have systematically studied the effects of baking on the sticking coefficient and this is shown in Fig. 4. The relevant conditions are shown in Table 1.

Table 1. Baking Conditions Pertaining to the Data in Fig. 4.

Curve	1000°C Baking	High Vacuum (Torr)
1	†Contaminated oven	1×10^{-7}
2	unbaked	1×10^{-7}
3	*Clean	1×10^{-7}
4	Clean	$\sim 1 \times 10^{-8}$

†The quartz baking oven had rubber stoppers. During the baking the stoppers released contaminants into the oven thereby poisoning the graphite getters.

*The oven was refurbished with quartz stoppers and tubes to reduce contamination.

2. Long Exposure Times

Our primary focus is to obtain reliable and reproducible data for the gettering efficiency of graphite continuously exposed to a Cs atomic beam for extended periods of time (~ days and months), and under conditions similar to that inside a CBT. This will enable us to develop a good theoretical model for gettering which can subsequently be used to specify and characterize the gettering efficiency of graphite in spaceborne clocks.

The variation of γ with time for three different types of graphite and an aquadag coated surface is shown in Fig. 5. The percentage porosity of the respective samples is also indicated in Fig. 5. The sudden steps in the data
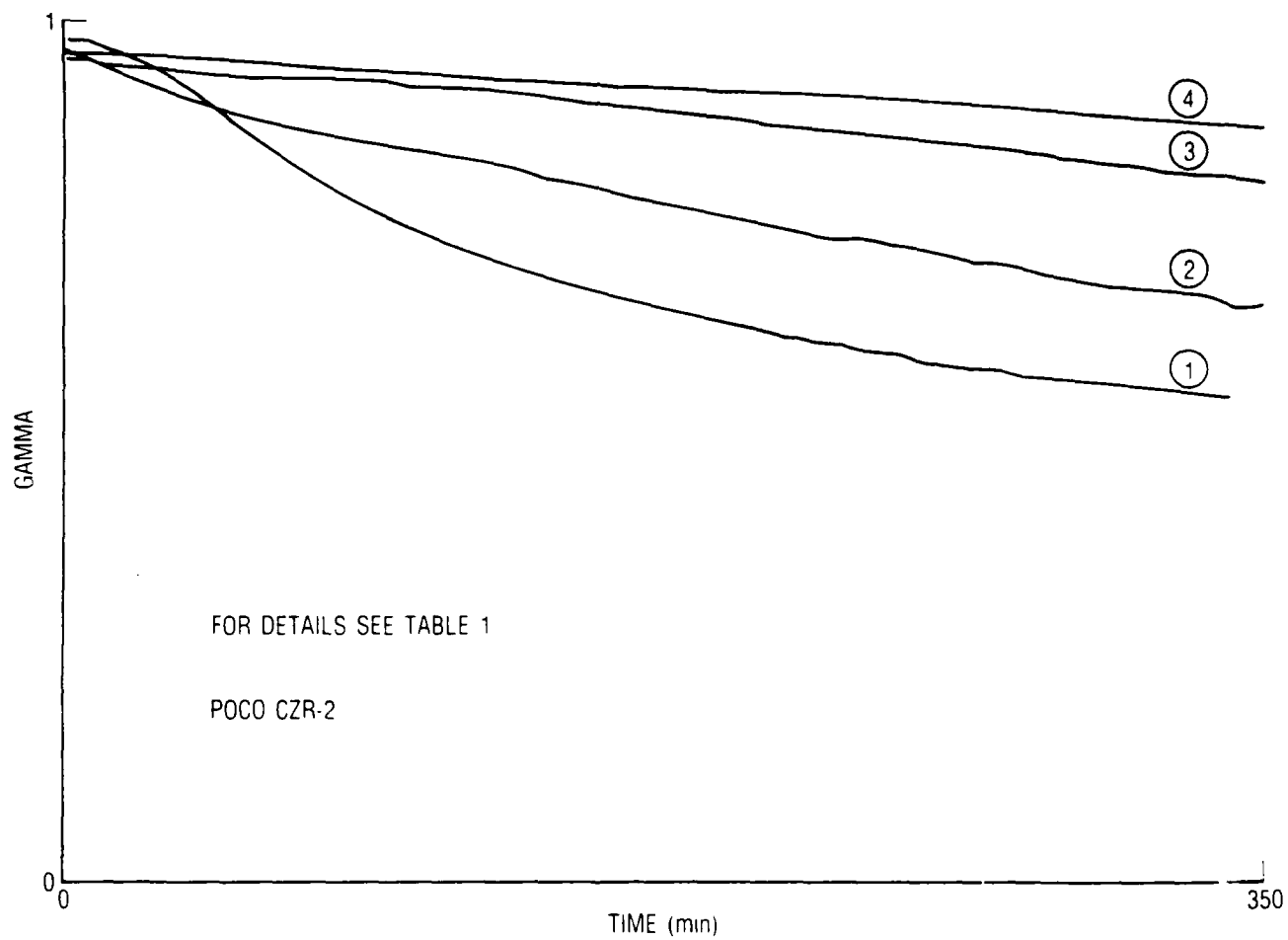
Fig. 4. Variation of γ with Time for Baked and Unbaked Graphite Targets. Baking is done at 1000°C as explained in the text. Data from Ref. 1 is also included.

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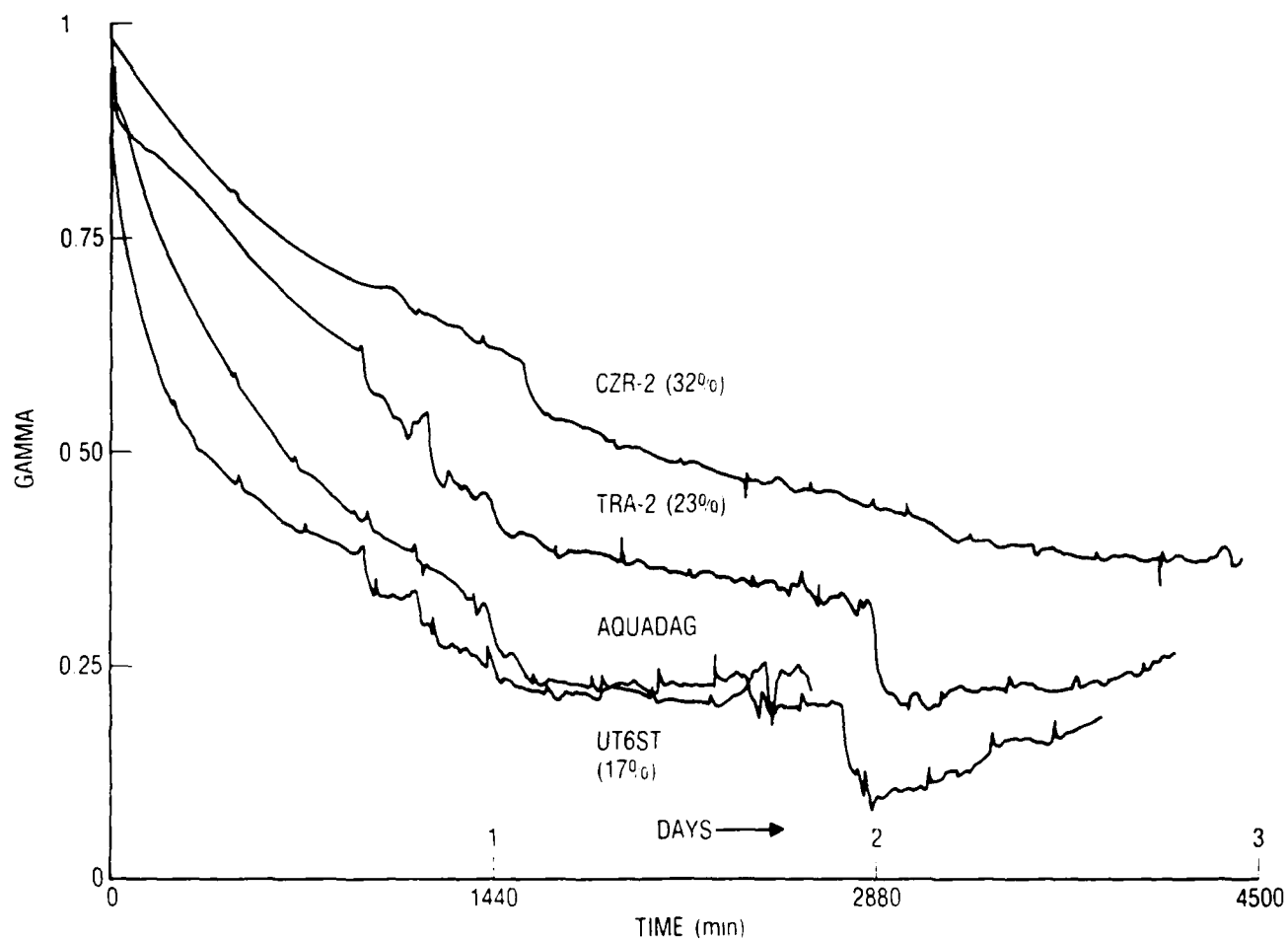
Fig. 5. Long term (60 - 75 h) Variation of the Sticking Coefficient as a Function of Time. At the end of 60-70 h of exposure to the Cs beam, the sticking coefficient drops to ~ 0.2-0.4, depending on the type of graphite. The Cs beam intensity used in these measurements is 4×10^2 atoms/cm^2 sec. The porosity of the different types of graphite is shown in parenthesis.

are due to fluctuations in the incident atomic beam which remains very stable only for about 24 hours. A differential measurement, with continuous monitoring of both the incident and the reflected beams, would be more desirable for these types of long term experiments. Suitable corrections for the beam fluctuation were made in computing γ.

Different types of graphite studied show similar behavior. γ starts with an initial value of 1 and decreases to about 0.2-0.4 after 60-75 hours of exposure. The Cs beam flux for these data runs was ~ 4 $\times 10^{12}$ atoms/cm^{2}sec. POCO CZR-2 exhibits a distinctly slower rate of decrease of γ than all the other types investigated.

III. BULK GETTERING STUDIES

A. PROCEDURE EMPLOYED IN THE EXPOSURE OF GRAPHITE TO CESIUM VAPOR

Four types of standard synthetic graphite along with Aquadag-coated
surfaces were exposed to Cs vapor. The graphites selected for study, POCO
CZR-2(32%), TRA-2 (23%), Ultra Carbon UT-6ST (17%), and UT-8 (17%), have high
porosities (the fractions of volume due to voids in graphite are shown in
parentheses) which we believe should yield superior gettering capabilities.
Graphite samples (0.5 in. × 0.5 in. × 0.125 in.) were fabricated using
greaseless machining tools. These samples weighed approximately 1g. Aquadag
was applied to cleaned stainless steel plates (1.0 cm × 1.0 cm × 0.16 cm).
Both the graphite and Aquadag samples were baked under liquid-nitrogen-trapped
mechanical pump vacuum at 500°C to remove any adsorbed water. The samples
were then transferred to glass manifolds of the design shown in Fig. 6. Each
graphite-containing manifold holds eight samples, two of each type of graphite
being studied. Prior to opening the Cs reservoir, the manifold is attached to
a glass vacuum system. On the vacuum system the manifolds are baked at 350°C
until the system pressure drops below 1×10^{-6} torr, typically 24 hours.
After baking, the Cs reservoir is opened and degassed at which point the
manifold, still evacuated, is removed from the vacuum system. Cs is then
transferred from the reservoir to other surfaces as indicated in Fig. 6. This
provides a source of Cs vapor with a large surface area. The manifolds are
placed in a 50°C oven and the graphite samples are exposed to Cs vapor for the
desired periods of time. In these studies exposure times ranged from six to
fifteen months, allowing the temporal evolution of Cs absorption to be
monitored.

We wished to determine how much Cs the graphite (or Aquadag) sample could
absorb. Consequently, it was essential to ensure that the observed Cs
absorption was due to the properties of the graphite and had not been limited
by the amount of available Cs vapor. With this in mind, a large surface area
of metallic Cs was employed so that copious amounts of Cs vapor would be
available. Also, the manifolds were fabricated such that there are no
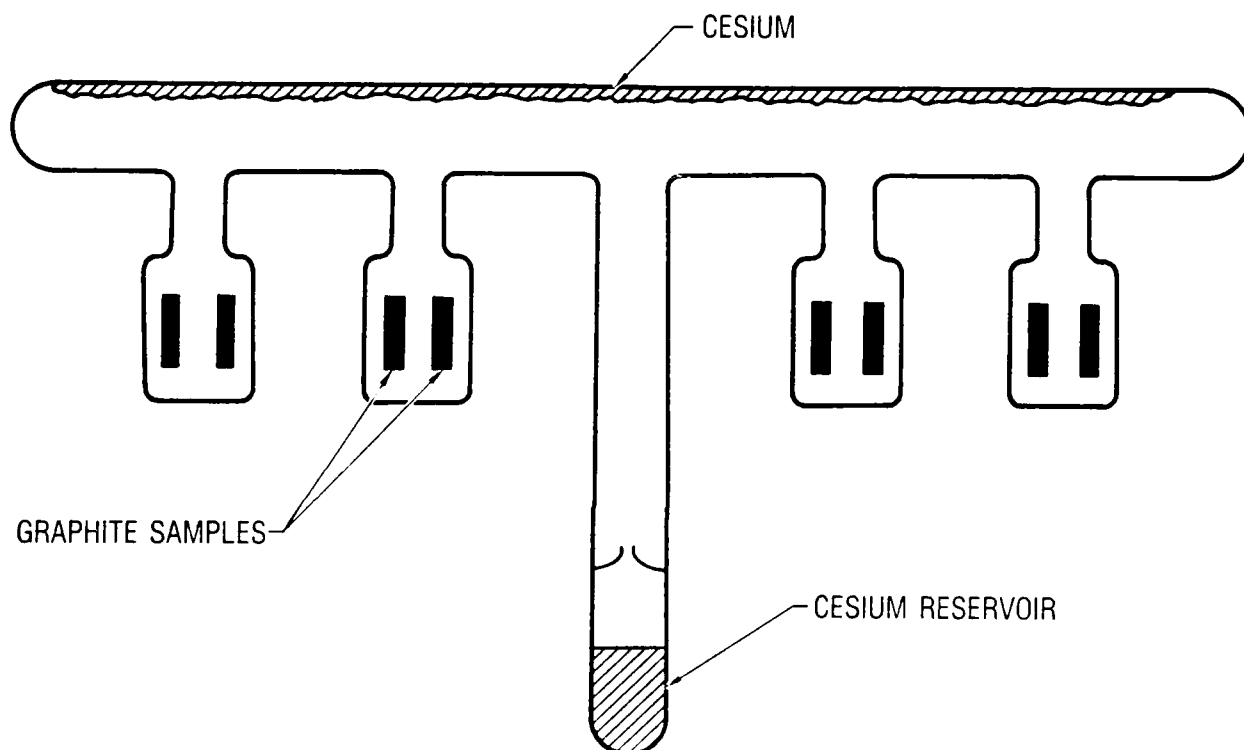
Fig. 6. Glass Manifold Used to Expose Graphite Samples to Cesium Vapor. After evacuation the manifold is placed in a 50°C oven.

serious conductance limitations on the amount of Cs vapor that could pass through the constrictions. If simplistically we assumed these atoms impact the graphite surfaces uniformly, the incident flux is on the order of 7×10^{13} atoms/cm^2 sec. This is close to the intensities seen by graphite collimators placed near the oven in GPS Cs beam tubes and larger than the intensities graphite surfaces would be exposed to in other locations.

B. DETERMINATION OF THE AMOUNT OF ABSORBED CESIUM

1. Gravimetric Procedures

After investigating several techniques, we have found that the most reliable method to determine the amount of Cs absorbed by a graphite sample is a simple gravimetric procedure. Prior to placement in the glass manifold graphite samples are accurately weighed. After Cs exposure the glass manifold is opened and the samples reweighed. The increase in sample weight is attributed to Cs absorption. Cs reacts with water and the reaction product, CsOH, is hygroscopic. As a result, care is taken to minimize exposure of the sample to water vapor during manifold opening and sample weighing. Additionally, experiments were performed to determine how rapidly Cs-impregnated graphite absorbs water. Graphite samples were placed on scales and the weight increases measured as a function of the exposure time to laboratory air. One example of these studies was a sample of POCO CZR-2 that showed a weight increase due to Cs exposure of 21%. After exposure to laboratory air for 30 minutes the weight increase had risen to 23%. After two hours of laboratory air exposure, the weight increase had further risen to 25.5%. These results actually have an uncertainty as they depend on the humidity of the laboratory air. Three manifolds were opened and the samples transfered to a desiccator within a glove bag containing dry nitrogen. Their only exposure to the laboratory atmosphere was during the weighing process. Our studies indicated that an insignificant amount of water is absorbed during the three to four minutes needed for weighing. Two additional manifolds containing graphite samples and a single manifold holding the Aquadagged samples were opened in air and the samples transferred to the desiccator. The water vapor absorption experiments indicate that a graphite sample's weight

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should increase by approximately three to five percent due to the water vapor-Cs reaction which might have taken place during the manifold opening. Consequently we consider the weight increases from the last three manifolds to be less reliable than those of the other three manifolds. The percentage weight increases of the graphite samples after Cs exposure are summarized in Table 2.

The sample-to-sample variations in weight are greater than the uncertainties associated with the weighing process. At this point, it is not clear whether the variations are indicative differences in the gettering abilities of various samples or result from a subtle systematic limitation of our exposure procedure. In spite of the variations, several observations may be made. It appears that the absorptive capability of graphite increases with increasing porosity. Based on the results of the manifolds opened under dry nitrogen, fractional increases in graphite weight resulting from Cs saturation may range from 4 to 20% depending on the graphite type. POCO CZR-2 graphite displayed the highest absorption capability (~ 20% weight increase). Aquadag-covered surfaces also displayed high absorption capabilities, approximately 30% by weight. This result may be artifically high as the aquadag manifold was opened in laboratory air. It must also be noted that these coatings have only limited mass per surface area and hence limited absolute absorptive ability. In our studies the coatings, resulting from a single application of the aquadag solution, displayed surface densities of approximately 10 mg/cm^2. This would indicate each square centimeter of painted surface could at most absorb about 3 mg of Cs. Using the information supplied in Table 2, manufacturers will be able to reasonably estimate the minimum graphite needed to absorb the initial Cs charge in the beam tube.

2.   Other Measurement Techniques

Several other techniques such as, scanning Auger, atomic absorption spectroscopy, and acid-base titrations, were applied to the measurement of Cs absorbed by graphite. Of these, the acid-base titration was simplest to perform and had the highest intrinsic accuracy. This procedure is based on the reaction between Cs and water,

Table 2. Summary of Gravimetric Measurements

	Manifolds Opened Under Dry Nitrogen					Manifolds Opened Under Laboratory Air		
EXPOSURE TIME (MONTHS)	6(1)	9	15	SATURATION LEVEL (2)	POROSITY (%)	6	10	9
GRAPHITE SAMPLE	Fractional Weight Gain (Acid-Base Titration Result) (%)					Fractional Weight Gain (Acid-Base Titration Result) (%)		
Ultra Carbon #1	3.1	4.2	1.6 (1.1)			16.9	25.0	
UT-6ST(3) #2	3.7	5.4	4.7	4 ± 2	17	11.4	17.7	
Ultra Carbon #1	4.7	9.5	6.7 (1.7)			7.9	16.1	
UT-8(4) #2	3.8	11.0	5.7	8 ± 2.5	17	10.	15.0	
POCO #1	4.2	12.2	12.1 (1.2)			6.9	8.1	
TRA-2 #2	2.6	8.1	10.5	11 ± 2	23	7.0	6.6	
POCO #1	5.2	23.5	17.9 (4.7)			17.9	14.2	
CZR-2 #2	3.5	8.0	19.9	20 ± 3	32	4.8	12.2	
Aqua-Dag #1								32
Coatings #2								33
#3								40
#4								26 (6.5)

(1) Graphite samples baked at 1100°C, all other samples baked at 500°C
(2) Average of all 9 and 15 month data except for CZR-2 in which 8% value was rejected on statistical grounds
(3) Grain size 0.04 mm
(4) Grain size 0.02 mm

$$Cs + H_2O \rightarrow CsOH + 1/2 \ H_2. \hspace{2cm} (1)$$

In an excess of water, the CsOH dissociates producing OH^- ions and, as a result, a normally neutral water solution becomes basic. For each dissolved Cs atom, an extra OH^- ion is produced. In the acid-base titration an amount of acidic solution, which supplies H^+ ions sufficient to neutralize the excess OH^-, is added to the basic solution. When neutrality is regained, the titration end point has been reached and the number of H^+ ions added will equal the initial number of dissolved Cs atoms. In performing this titration, one hopes to obtain a completely independent measure of the amount of Cs absorbed by graphite. This measurement will also be insensitive to any water vapor absorbed by the graphite sample.

After weighing, several graphite samples taken from the fifteen month manifold were crushed and soaked in distilled water. Using hydrochloric acid solutions, with precise hydrogen ion concentrations, the titrations were performed. To identify the titration end-point, a phenolphthalein indicator was added to the solution. When neutrality is approached this indicator undergoes a dramatic color change. Comparison of the amounts of absorbed Cs indicated by the titration with that of the gravimetric analysis is also given in Table 2. The titrations consistently show lower amounts of absorbed Cs, approximately 10 to 25% of that found by the gravimetric technique. A requirement for the acid-base titration to be accurate is that all of the absorbed Cs atoms undergo reaction (Eq. 1). Apparently, a major portion of the absorbed Cs is not readily dissolved in water. This finding is also consistent with our atomic absorption measurements that detect atomic Cs. Prior to the absorption measurement, the absorbed Cs must be similiarly dissolved in a water solution. All atomic absorption measurements indicated Cs absorption was less than that found by the gravimetric analysis, typically only approximately 10% of the gravimetric result. These findings give insights into the Cs absorption process and will be discussed subsequently.

A final comment concerns scanning Auger measurements made upon Cs-exposed graphite samples. Auger measurements produce signals proportional to the Cs

atom concentration in the graphite. Unfortunately, it is very difficult to obtain reliable absolute concentrations. The Auger technique was applied to cross sections of graphite samples, giving valuable penetration profile information but not absolute concentrations.

IV. DISCUSSION AND CONCLUSIONS

Our experimental results represent the _first_ _quantitative_ investigation of the Cs gettering capabilities of graphite used in beam tube standards. We have demonstrated the feasibility of making long term studies of the gettering properties of graphite under conditions similar to that encountered in Cs beam tubes. As expected γ decreases with time. The most surprising aspect of our data is the rapid decrease in γ from the initial value of 1 to about 0.2-0.4 after just a few days of exposure to the Cs atomic beam. Undoubtedly γ will continue to decline and eventually will be zero. The fact that γ, after just a few days of exposure, is merely 0.2-0.4 has implications on the long term tube reliability and provides insights into how gettering occurs within the CBT. To put things in perspective, we compared the actual Cs beam flux that the graphite block placed in the vicinity of the oven is exposed to with the beam flux that we have used in our studies. For graphite blocks placed at a distance of 2 to 3 mm from the oven, the effective Cs beam flux can be much more than an order of magnitude larger than the flux used in our experiments. Our studies indicate that more than 60% of the Cs atoms incident on this graphite block will not stick. This would imply that, at the very early life of the beam tube, this block of graphite would lose most of its gettering capabilities. This would also mean that a large part of the gettering would have to be efficiently done by all the graphite coated surfaces and graphite blocks placed at other locations.

Our bulk gettering data provide for the first time a quantitative way of estimating how much graphite should be placed in a beam tube to getter the initial Cs charge of the oven. This is particularly important for Cs beam tubes, such as those used on GPS satellites, upon which stringent reliability requirements are placed. Estimates of the amount of graphite needed in a beam tube based on our results should be considered absolute minimum amounts. The potential sample-to-sample variation in absorptive capability of a given graphite indicates that not all pieces may be able to absorb the same amount of Cs. Also, as a getter piece begins to approach Cs saturation, the behavior

of the sticking coefficient is not known. If the sticking coefficient is
extremely low, the Cs background pressure could reach deleterious levels prior
to complete saturation. Both of these concerns require further experiments
and analysis to determine their importances.

A difficult problem to address experimentally is getter performance after
a number of years of Cs exposure. Theoretical approaches may be the most
productive when analyzing long term gettering and, in particular, sticking
coefficient evolution. While a rigorous model of Cs gettering by graphite
does not yet exist, the present experiments are of value in arriving at simple
models. Graphite is composed of coke grains which are held together with
pitch residue. Cs atoms that stick to the graphite surface must penetrate
into the graphite or the sticking coefficient would rapidly go to zero. The
penetration of Cs into graphite is typically treated as a "grain boundary"
process. (Ref. 3) Processes of this type are characterized by two diffusion
mechanisms proceeding at dramatically different rates. Between the grains
diffusion proceeds rapidly. Penetration into the grains proceeds at a much
slower rate. However, due to the large grain surface area, a major fraction
of Cs should be able to penetrate into the grains in reasonable periods of
time. This is consistent with results of the acid-base titration that
indicated 70-90% of the absorbed Cs could not be dissolved in water.
Apparently this Cs had penetrated into the grains. At present we are using
bulk exposure and sticking coefficient data to develop a theoretical model of
the gettering process. This should allow accurate prediction of sticking
coefficient behavior even after many years of Cs exposure.

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Computer Science Laboratory: Program verification, program translation, performance-sensitive system design, distributed architectures for spaceborne computers, fault-tolerant computer systems, artificial intelligence, micro-electronics applications, communication protocols, and computer security.

Electronics Research Laboratory: ..croelectronics, solid-state device physics, compound semiconductors, radiation hardening; electro-optics, quantum electronics, solid-state lasers, optical propagation and communications; microwave semiconductor devices, microwave/millimeter wave measurements, diagnostics and radiometry, microwave/millimeter wave thermionic devices; atomic time and frequency standards; antennas, rf systems, electromagnetic propagation phenomena, space communication systems.

Materials Sciences Laboratory: Development of new materials: metals, alloys, ceramics, polymers and their composites, and new forms of carbon; non-destructive evaluation, component failure analysis and reliability; fracture mechanics and stress corrosion; analysis and evaluation of materials at cryogenic and elevated temperatures as well as in space and enemy-induced environments.

Space Sciences Laboratory: Magnetospheric, auroral and cosmic ray physics, wave-particle interactions, magnetospheric plasma waves; atmospheric and ionospheric physics, density and composition of the upper atmosphere, remote sensing using atmospheric radiation; solar physics, infrared astronomy, infrared signature analysis; effects of solar activity, magnetic storms and nuclear explosions on the earth's atmosphere, ionosphere and magnetosphere; effects of electromagnetic and particulate radiations on space systems; space instrumentation.

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